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(71) Applicant(s)
Tetronics Limited
(Incorporated in the United Kingdom)
5 Lechdale Road, FARINGDON, Oxfordshire, SN7 9AJ, United Kingdom

(72) Inventor(s)
David Edward Deegan
Christopher David Chapman
Timothy Paul Johnson

(74) Agent and/or Address for Service
Boult Wade Tennant
Verulam Gardens, 70 Gray's Inn Road, London, WC1X 8BT, United Kingdom

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(56) Documents Cited
GB 2273027 A **EP 0766502 A1** **US 5420391 A**
US 5408066 A **US 5332885 A** **US 4877937 A**
US 4238427 A **US 3989512 A**
WPI Accession no 96-480689 & JP080243756 A
WPI Accession no 95-012138 & JP060299209 A
WPI Accession no 92-013623 & JP030264601 A

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INT CL[7] **B22F 9/00 9/02 9/04 9/14 , H05H 1/26 1/34 1/42**
Online: WPI, EPODOC, JAPIO

(54) Abstract Title
Plasma production of fine powders using an electrode with a channel

(57) A plasma arc reactor for producing a powder from a solid feed material, for example aluminium, is provided comprises:
(a) a first electrode 5,
(b) a second electrode 10 which is adapted to be spaced apart from the first electrode by a distance sufficient to achieve a plasma arc therebetween,
(c) means for introducing a plasma gas into the space between the first and second electrodes,
(d) means for generating a plasma arc in the space between the first and second electrodes,
wherein the first electrode has a channel 7 running therethrough, an outlet of the channel exiting into the space between the first and second electrodes, and wherein means are provided for feeding solid material through the channel to exit therefrom via the outlet into the space between the first and second electrodes.

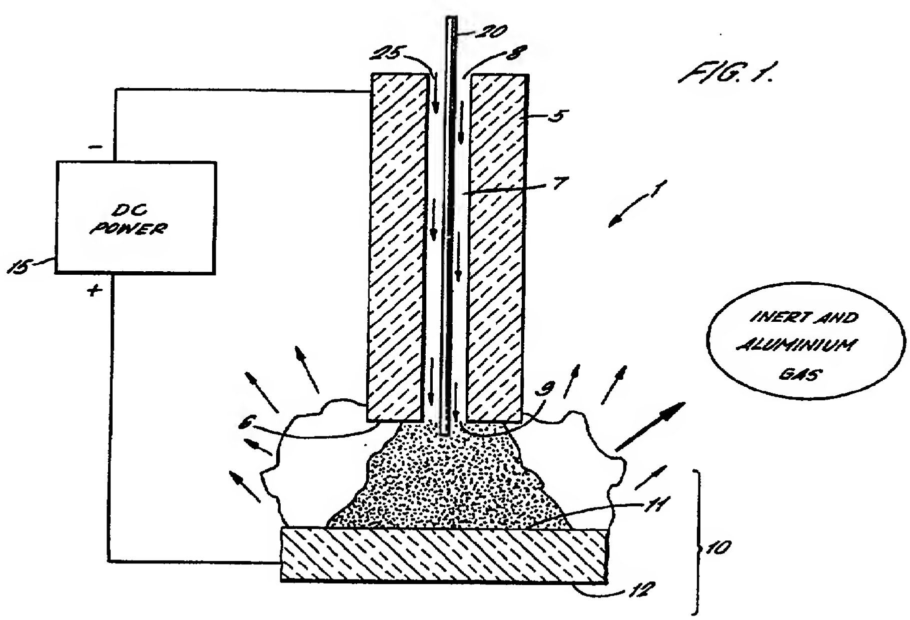


FIG. 1.

GB 2 359 096 A

At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

FIG. 1.

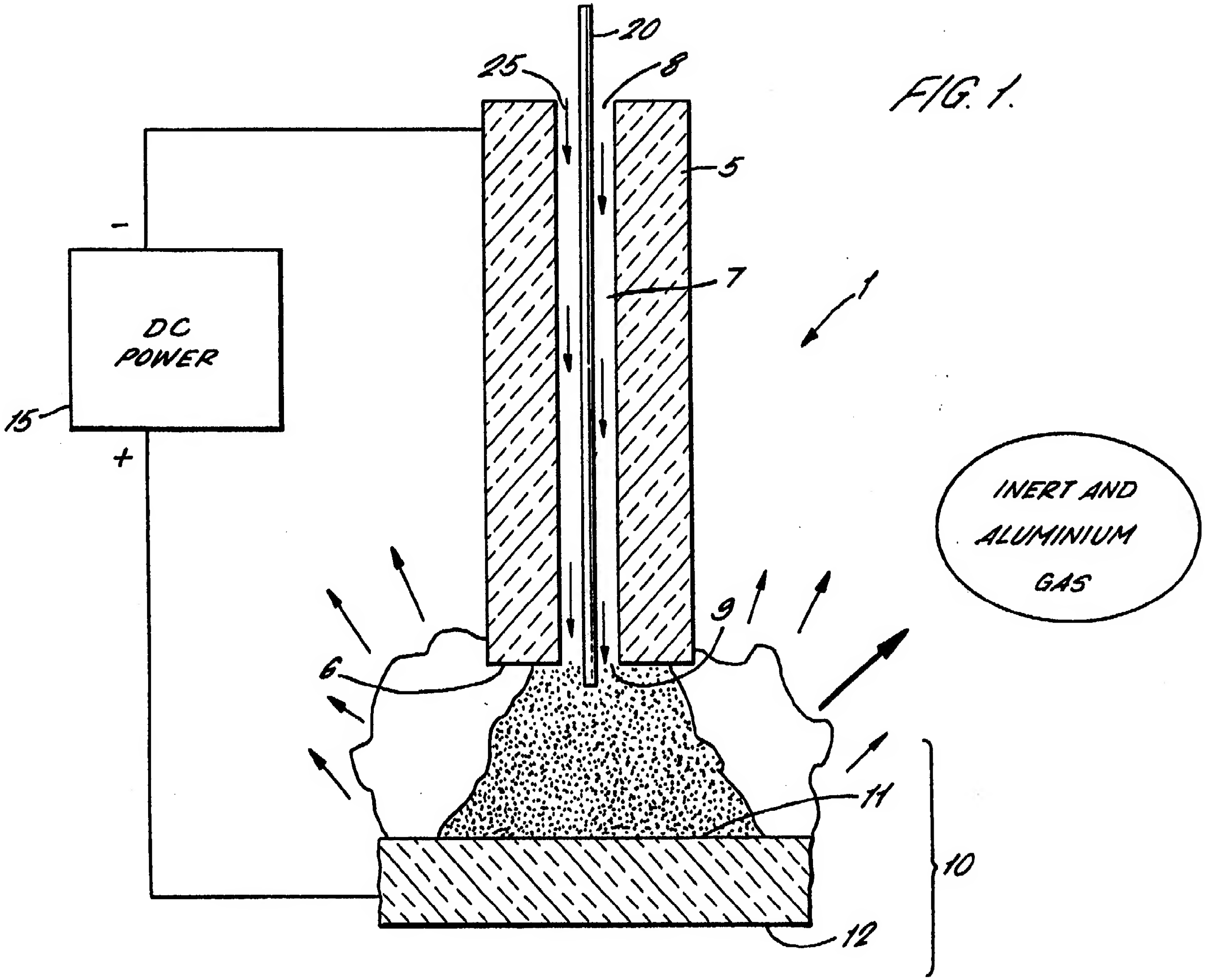
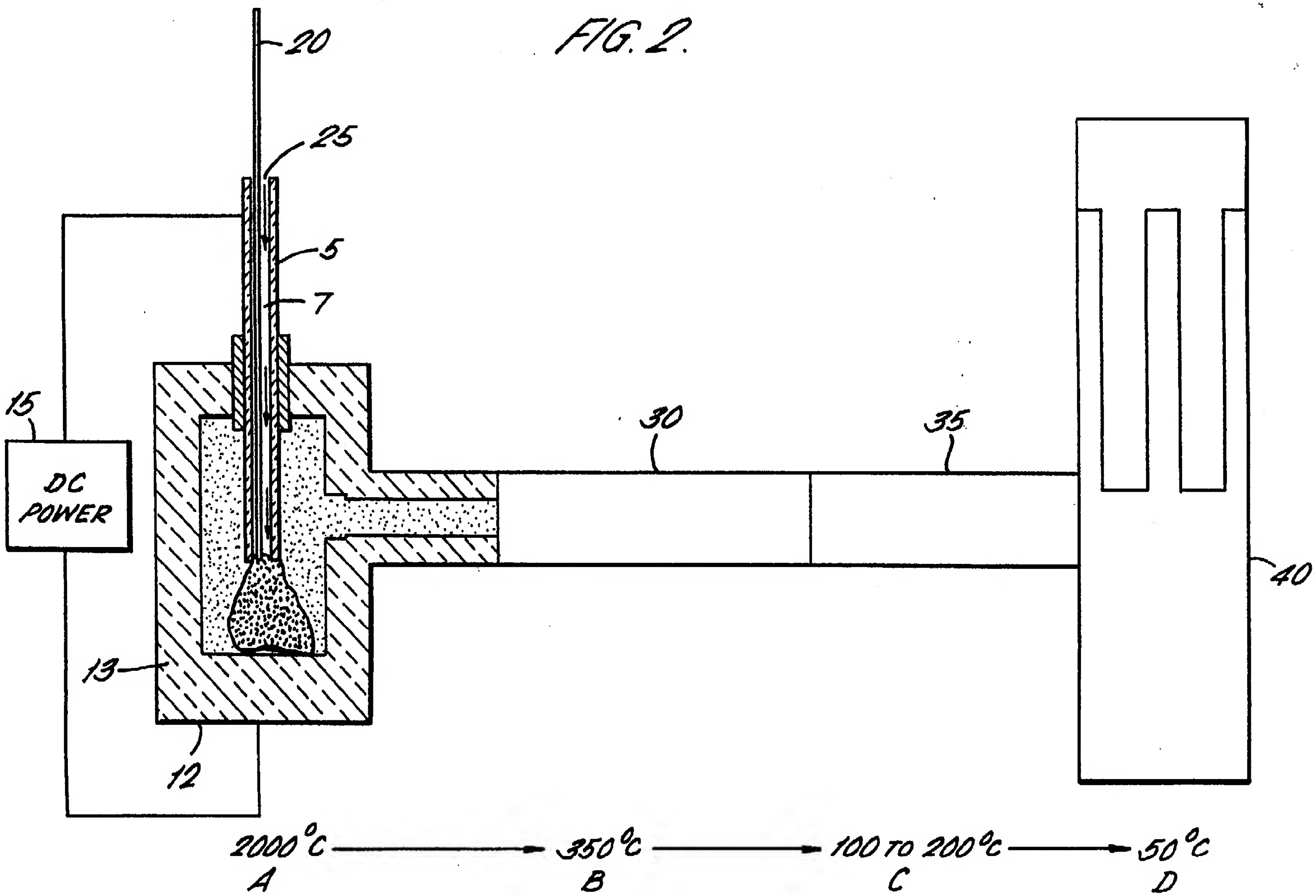


FIG. 2.



20
25
5
7
15
DC POWER
13
12
30
35
40
2000°C A
350°C B
100 TO 200°C C
50°C D

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Apparatus and process for the production of fine powders

The present invention relates to an apparatus and process for the production of fine powders. In particular, a plasma arc reactor is provided which may be used in a plasma evaporation process to produce ultra-fine (i.e. sub-micron) aluminium powders.

Metal and ceramic powders are used in sintering processes in metallurgy and in catalysis in the chemical industry. The powders may be used in the manufacture of structural components, magnetic films, chemical coatings or as oil additives.

The present invention provides a plasma arc reactor for producing a powder from a solid feed material, the reactor comprising:

(a) a first electrode,

(b) a second electrode which is adapted to be spaced apart from the first electrode by a distance sufficient to achieve a plasma arc therebetween,

(c) means for introducing a plasma gas into the space between the first and second electrodes,

(d) means for generating a plasma arc in the space between the first and second electrodes,

wherein the first electrode has a channel running therethrough, an outlet of the channel exiting into the space between the first and second electrodes, and wherein means are provided for feeding solid material into and through the channel to exit therefrom via the

product is preferably then sealed, in inert gas, in a container at a pressure above atmospheric pressure.

The channel in the first electrode is advantageously adapted to additionally introduce the plasma gas into the space between the first and second electrodes. Thus, the solid feed material and plasma gas may travel through a common channel and exit the electrode via a common outlet into the space between the first and second electrodes.

The means for generating a plasma arc in the space between the first and second electrodes will generally comprise a DC or AC power source.

If desired, one or more additional electrodes, also having a material feed channel therein, may be used to enable different materials to be co-fed into a single plasma reactor. A common counter electrode may be used or, alternatively, separate counter electrodes may be provided, each opposing an electrode with a channel therein. Common or separate power supplies may be used, although separate power supplies are preferred since this allows different evaporation rates for different materials.

The apparatus according to the present invention may operate without using any water-cooled elements inside the plasma reactor and allows replenishment of solid feed material without stopping the reactor.

The present invention also provides a process for producing a powder from a solid feed material, which process comprises:

(i) providing a plasma arc reactor as herein described,

electrode, may take any appropriate form to enable a plasma arc to be generated between it and the first electrode. The second electrode may simply have a substantially planar arc portion. For example, the second electrode may be disposed as a planar substrate on the bottom wall of the plasma reactor.

The arc portions of the first and/or second electrodes will generally be formed from graphite.

The plasma reactor may be provided in the form of a graphite lined vessel or a graphite crucible, in which a portion thereof acts as the second electrode. Accordingly, the second electrode may be integrally formed with the reactor vessel.

The plasma arc reactor advantageously further comprises cooling means for cooling and condensing solid material which has been vaporised in the plasma arc generated between the first and second electrodes. The cooling means preferably comprises a source of a cooling gas.

The second electrode preferably comprises a graphite vessel having a surface adapted to direct vaporised material downstream to a cooling zone to be cooled, in use, by the cooling gas.

A collection zone may be provided downstream of the cooling zone for collecting a powder of the condensed vaporised material. The collection zone may comprise a filter cloth which separates the powder particulate from the gas stream. The filter cloth is preferably mounted on an earthed cage to prevent electrostatic charge build up. The powder may then be collected from the filter cloth, preferably in a controlled atmosphere zone. The resulting powder

At least some cooling of the vaporised material may be achieved using an inert gas stream, for example argon and/or helium. Alternatively, or in combination with the use of an inert gas, a reactive gas stream may be used. The use of a reactive gas enables oxide and nitride powders to be produced. For example, using air to cool the vaporised material can result in the production of oxide powders, such as aluminium oxide powders. Similarly, using a reactive gas comprising, for example, ammonia can result in the production of nitride powders, such as aluminium nitride powders. The cooling gas may be recycled via water-cooled conditioning chamber.

The surface of the powder may be oxidised using a passivating gas stream. This is particularly advantageous when the material is aluminium or aluminium-based. The passivating gas may comprise an oxygen-containing gas, and a particularly preferred gas comprises from 95 to 99 vol.% of an inert gas, such as helium and/or argon, and from 1 to 5 vol.% of oxygen, more preferably approximately 98 vol.% of the inert gas(es) and approximately 2 vol.% of oxygen. Such a gas mixture has been found to produce particularly good results for aluminium and aluminium-based materials. The (inert) cooling gas may be recycled and subsequently diluted with oxygen at a rate of typically 1 NM^3/hour to provide the passivating gas stream. The reactivity of some ultra-fine powders presents an operational risk if there is a likely-hood of contact with, for example, water and/or air. The passivation stage renders the powdered material more suitable for transporting

The process according to the present invention may be used to produce a powdered material, such as aluminium, substantially all of the particles of which

(ii) introducing a plasma gas into the space between the first and second electrodes,

(iii) generating a plasma arc in the space between the first and second electrodes,

(iv) feeding solid material through the channel to exit via the outlet thereof into the plasma arc, whereby the solid feed material is vaporised,

(v) cooling the vaporised material to condense a powder, and

(vi) collecting the powder.

The solid feed material will generally comprise or consist of a metal, for example aluminium or an alloy thereof. The solid feed material may be provided in any suitable form which allows it to be fed into and through the channel to exit therefrom into the space between the electrodes. For example, the material may be in the form of a wire, fibres and/or a particulate.

The plasma gas will generally comprise or consist of an inert gas, for example helium and/or argon.

The plasma gas is advantageously injected into the channel in the first electrode to exit therefrom into the space between the first and second electrodes. In this case, the plasma gas and solid material preferably exit the first electrode via a common outlet. The plasma gas and solid material may be fed into the channel in the first electrode via a common inlet or, alternatively, via separate inlets. During operation, the plasma gas and solid material will be co-fed into the channel.

water above atmospheric pressure.

If a cooling gas such as argon or helium is used to cool and condense the vaporised material, a flow rate of from 60 to 120 Nm^3/h has been found to result in an aluminium powder in which most, if not substantially all, of the particles have a diameter of less than 200 nm in diameter (more typically ≤ 100 nm). After cooling, the gas and particulate temperature will typically be from 300 to 350°C.

The present invention will now be described further, by way of example, with reference to the accompanying drawings in which:

Figure 1 shows one embodiment of an electrode configuration which may be used in a plasma arc reactor according to the present invention.

Figure 2 provides a flow diagram of a process according to the present invention.

In Figure 1, a first electrode 5 is provided in the form of a cylindrical graphite rod which terminates at an arc tip 6. If desired, the upper portion of the graphite electrode 5 may be replaced with copper. The electrode 5 has a central bore formed therein which extends along the length of the electrode 5. The surface of the bore defines a closed channel 7 (or passageway) having an inlet 8 at one end and an outlet 9 disposed at the arc tip 6.

A second counter electrode 10 is provided as part of a graphite-lined reactor vessel (13) (see Figures 1 and 2). Only an arc portion 11 on the interior surface of the bottom wall 12 of the vessel 13 is shown in Figure 1. The whole of the vessel 13 is

having a diameter of less than 200 nm. Preferably the average particle diameter lies in the range of from 50 to 200 nm, more preferably from 50 to 100 nm.

It will be appreciated that the processing conditions, such as material and gas feed rates, temperature and pressure, will need to be tailored to the particular material to be processed and the desired size of the particles in the final powder.

It is generally preferable to pre-heat the reactor before vaporising the solid feed material. The reactor may be preheated to a temperature of at least about 2000°C and typically approximately 2200°C. Pre-heating may be achieved using a plasma arc.

The rate at which the solid feed material is fed into the channel in the first electrode will affect the product yield and powder size. When using aluminium wire, a feed rate of from 1 to 5 kg/hour has been used, more typically approximately 2 kg/hour.

The inert plasma gas, for example helium, may also be injected through the channel in the first electrode at a rate of from 2.4 to 6 Nm^3/h, more typically approximately 3 $Nm^3/hour$.

If a DC power supply is used to generate the plasma arc, then the DC amperage will generally be set at a value in the range of from 400 to 800 A. Typical DC Electrical characteristics are of the order of 800 A and between 30 to 40 V with a plasma arc column length of between 60 mm and 70 mm.

The plasma arc reactor according to the present invention is typically operated in excess of 750 mm of

inlet 8. The plasma gas 25 may be stored by conventional means in a gas tank, and controlled injection into the inlet may be achieved by the use of a valve. Accordingly, the feed rates of both the wire and the plasma gas may be controlled.

In use, the graphite-lined vessel 10 is preheated to a temperature of at least about 2000°C (typically approximately 2200°C) using the plasma arc. This entails injecting an inert plasma gas 25 through channel 7 in the first electrode 5 and switching on the power supply 15.

The reactor is typically operated in excess of 750 mm of water above atmospheric pressure.

Once the reactor has been pre-heated, aluminium wire 20 is then fed into the inlet 8 of channel 7 in the first electrode 5 at a rate of typically 2 kg/hour. Inert plasma gas is also injected through channel 7, typically at a rate of from 2.4 and 6 Nm^3/h, more typically approximately 3 Nm^3/hour.

Typical DC electrical characteristics are of the order of 800 A and from 30 to 40 V with a plasma arc column length of from 60 mm and 70 mm.

In this manner, the aluminium wire 20 is vaporised in the hot plasma gas (step A in Figure 2). The wire 20 and plasma gas 25 are continually fed into the channel 7 of the first electrode 7 as the wire 20 is vaporised in the plasma arc. Eventually a steady-state will be achieved. It will be appreciated that the feed rates of the wire 20 and/or gas 25 may be adjusted during processing.

The vaporised aluminium and plasma hot gas exits

shown in Figure 2 and it can be seen that the counter electrode forms an integral part of the reactor vessel 13. The arc portion 11 of the second electrode 10 opposes the arc tip 6 of the first electrode 5.

The first 5 and second 10 electrodes are connected to a DC power supply 15. The first electrode 5 is the cathode and the second electrode is the anode 10, although it will be appreciated that the polarities may be reversed.

The first electrode 5 is moveable with respect to the second electrode 10 and hence may be lowered to contact at the arc tip 6 thereof with the arc portion 11 of the second electrode 10 to complete the electrical circuit. The DC amperage from power supply 15 will generally be set at a value from 400 to 800 A. By raising the first electrode 5, a DC plasma arc can be established between the arc tip 6 of the first electrode 5 and the arc portion 11 of the second electrode 10.

A solid feed material, for example aluminium wire 20, can be fed into the inlet 8, to pass down the channel 7, out the outlet 9 and into the space between the arc tip 6 of the first electrode 5 and the arc portion 11 of the second electrode 10. An inert plasma gas 25, such as argon and/or helium, may similarly be injected through the channel 7, via the inlet 8, to exit the first electrode 5 at outlet 9. Accordingly, both the aluminium wire 20 and the plasma gas 25 may enter the first electrode 5 via a common inlet 8 and exit the electrode 5 via a common outlet 9 at the arc tip 6.

The wire 20 may be stored by conventional means on a coil or reel and fed by a multi-speed motor into

The resulting powder product is preferably then sealed, in inert gas, in a container at a pressure above atmospheric pressure.

If desired, one or more additional electrodes having a channel therein may be used to co-feed different metals into a single plasma vessel to produce, for example, alloy powders, sub-micron and nano-sized mixtures, oxides and nitrides. A common counter electrode may be used or, alternatively, separate counter electrodes may be provided, each opposing an electrode with a channel therein. Common or separate power supplies may be used, although separate power supplies are preferred since this allows for different evaporation rates for different metals.

The apparatus and process according to the present invention provide a simplified technique for the production and collection of ultra-fine, sub-micron powders. In a preferred embodiment, a transferred plasma arc is established between the arc tip of an elongate graphite electrode and a counter electrode formed as part of a graphite reactor crucible.

The apparatus according to the present invention may operate without using any water-cooled elements inside the plasma reactor and allows replenishment of feed material without stopping the reactor.

The reactivity of ultra-fine metals, such as aluminium, presents an operational risk if there is a likely-hood of contact with water, reactive liquids, or reactive gases such as air and oxygen. The passivation stage described herein renders the powdered material more suitable for transporting.

the reactor vessel under the influence of the gas being injected through the channel 7 in the first electrode 5. The vaporised aluminium is then quenched in a cooling zone 30 using an inert cooling gas stream, such as argon or helium, to condense a sub-micron powder of aluminium (step B in Figure 2). The flow rate of the cooling gas stream is typically from 60 to 120 Nm^3/h, and the particles of the aluminium powder are typically from ≤ 200 nm in diameter (more typically ≤ 100 nm). After the inert gas quench, the gas and particulate temperature is typically from 300 to 350°C.

If desired, a passivation step may next be carried out in a passivation zone 35 downstream of the cooling zone 30 (step c in Figure 2). This may be achieved in a number of ways. The cooling gas may be recycled to a water-cooled conditioning chamber for further cooling, and then injected back into the apparatus, together with up to 5 vol.% of oxygen to contact with the powder. Typically, the oxygen is introduced at a rate of approximately 1 Nm^3/h. Alternatively, a separate source of the passivation gas may be used. The temperature during the passivation step is typically in the range of from 100 to 200°C.

After the passivation step, the powder particulate and gas stream pass to a collection zone 40 which contains a filter cloth (not shown) to separate the particulate from the gas (see step D in Figure 2). The filter cloth is preferably mounted on an earthed cage to prevent electrostatic charge build up. The gas may be recycled.

The powder may then be collected from the filter cloth, preferably in a controlled atmosphere zone.

channel, the elongate member terminating at an arc tip which opposes the second electrode, wherein the outlet of the closed channel is disposed at or adjacent to the arc tip.

4. A plasma arc reactor as claimed in any one of the preceding claims, wherein arc portions of the first and/or second electrodes is/are formed from graphite.

5. A plasma arc reactor as claimed in any one of the preceding claims, further comprising cooling means for cooling and condensing solid feed material which, in use, has been vaporised in the plasma arc between the first and second electrodes.

6. A plasma arc reactor as claimed in claim 5, wherein the cooling means comprises a source of a cooling gas.

7. A plasma arc reactor as claimed in claim 6, wherein the second electrode comprises a graphite vessel having a surface adapted to direct vaporised solid material downstream to a cooling zone to be cooled, in use, by the cooling gas.

8. A plasma arc reactor as claimed in any one of the preceding claims, further comprising a collection zone for collecting powdered material.

9. A plasma arc reactor as claimed in any one of the preceding claims, wherein the channel in the first electrode is also adapted to introduce the plasma gas into the space between the first and second electrodes.

10. A plasma arc reactor as claimed in any one of the preceding claims, wherein the means for generating a

CLAIMS:

1. A plasma arc reactor for producing a powder from a solid feed material, the reactor comprising:

(a) a first electrode,

(b) a second electrode which is adapted to be spaced apart from the first electrode by a distance sufficient to achieve a plasma arc therebetween,

(c) means for introducing a plasma gas into the space between the first and second electrodes,

(d) means for generating a plasma arc in the space between the first and second electrodes,

wherein the first electrode has a channel running therethrough, an outlet of the channel exiting into the space between the first and second electrodes, and wherein means are provided for feeding solid material through the channel to exit therefrom via the outlet into the space between the first and second electrodes.

2. A plasma arc reactor as claimed in claim 1, wherein the first electrode is moveable with respect to the second electrode from a first position at which an arc portion thereof contacts with an arc portion of the second electrode to a second position at which said arc portions are spaced apart from each other by a distance sufficient to achieve a plasma arc therebetween.

3. A plasma arc reactor as claimed in claim 1 or claim 2, wherein the first electrode is a hollow elongate member whose inner surface defines a closed

inert gas.

16. A process as claimed in claim 15, wherein the plasma gas comprises or consists of helium and/or argon.

17. A process as claimed in any one of claims 11 to 16, wherein the plasma gas is injected through the channel of the first electrode to exit therefrom into the space between the first and second electrodes.

18. A process as claimed in claim 17, wherein the plasma gas and solid feed material exit the first electrode via a common outlet.

19. A process as claimed in claim 17 or claim 18, wherein the plasma gas and solid feed material enter the channel in the first electrode via a common inlet.

20. A process as claimed in any one of claims 11 to 19, wherein at least some cooling of the vaporised material is achieved using an inert gas stream.

21. A process as claimed in any one of claims 11 to 20, wherein at least some cooling of the vaporised material is achieved using a reactive gas stream.

22. A process as claimed in any one of claims 11 to 21, wherein the surface of the powder is oxidised using a passivating gas stream.

23. A process as claimed in claim 22, wherein the passivating gas comprises an oxygen-containing gas.

24. A process as claimed in claim 23, wherein the oxygen-containing gas comprises from 95 to 99 vol.% of an inert gas and from 1 to 5 vol.% of oxygen.

plasma arc in the space between the first and second electrodes comprises a DC or AC power source.

11. A process for producing a powder from a solid feed material, which process comprises:

(i) providing a plasma arc reactor as defined in any one of the preceding claims,

(ii) introducing a plasma gas into the space between the first and second electrodes,

(iii) generating a plasma arc in the space between the first and second electrodes,

(iv) feeding solid material through the channel to exit via the outlet thereof into the plasma arc, whereby the solid feed material is vaporised,

(v) cooling the vaporised material to condense a powder, and

(vi) collecting the powder.

12. A process as claimed in claim 11, wherein the solid feed material comprises or consists of a metal or alloy.

13. A process as claimed in claim 12, wherein the solid feed material is aluminium or an alloy thereof.

14. A process as claimed in any one of claims 11 to 13, wherein the solid feed material is in the form of a wire, fibres and/or a particulate.

15. A process as claimed in any one of claims 11 to 14, wherein the plasma gas comprises or consists of an

25. A process as claimed in claim 24, wherein the oxygen-containing gas comprises approximately 98 vol.% of an inert gas and approximately 2 vol.% of oxygen.

26. A process as claimed in any one of claims 11 to 25, wherein the powder comprises particles substantially all of which have a diameter of less than 200 nm.

27. A plasma arc reactor for producing a powder from a solid feed material substantially as hereinbefore described with reference to or as illustrated in any one of the accompanying drawings.

28. A process for producing a powder from a solid feed material as claimed in 11, substantially as hereinbefore described.

Application No: GB 0003081.7
Claims searched: 1-28
Examiner: Pete Beddoe
Date of search: 30 May 2001

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.S): C7X X1; H1D (DGQ, DGQG)

Int Cl (Ed.7): B22F (9/00, 9/02, 9/04, 9/14); H05F (1/26, 1/34, 1/42)

Other: Online: WPI, EPODOC, JAPIO

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	GB 2273027 A	(UKAEA) see esp p2 line 20 - p5 line 20	1 at least
X	EP 0766502 A1	(SULZER) see esp col6 line 5 - col9 line 30 & figs 1,2	1,11 at least
X	US 5420391	(METCON) see esp col3 line 41 - col4 line 44 & fig 1	1,11 at least
X	US 5408066	(TRAPANI) see esp col2 line 51 - col3 line 29 & fig 3	1,11 at least
X	US 5332885	(LANDES) see esp col5 line 66 - col7 line 17 & figs	1,11 at least
X	US 4877937	(CASTOLIN) see esp col6 lines 47-55 & fig 3	1,11 at least
X	US 3989512	)NRDC) see esp col6 lines 47-55 & fig 3	1,11 at least
X	US 4238427	(CHISHOLM) see esp cols 2-3 & fig	1 at least
X	WPI Accession no 96-480689 & JP 8243756 A (MITSUBISHI) see abstract		1,11 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

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INVESTOR IN PEOPLE

Application No: GB 0003081.7
Claims searched: 1-28

Examiner: Pete Beddoe
Date of search: 30 May 2001

Category	Identity of document and relevant passage	Relevant to claims
X	WPI Accession no 95-012138 & JP 6299209 A (SANSHA) see abstract	1,11 at least
X	WPI Accession no 92-013623 & JP 3264601 A (DAIDO) see abstract	1,11 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
		E	Patent document published on or after, but with priority date earlier than, the filing date of this application.
&	Member of the same patent family		